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Indian Standard

METHODS OF SAMPLING AND TEST FOR ION-EXCHANGE RESINS

(First Revision)

First Reprint AUGUST 1997

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 7 *July* 1989

Indian Standard

METHODS OF SAMPLING AND TEST FOR ION-EXCHANGE RESINS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 29 July 1988, after the draft finalized by the Organic Chemicals (Misc) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Ion-exchange resins find use in water softening and de-ionization; milk softening (substitution of sodium ions for calcium ions in milk); removal of iron from wine and hydrochloric acid, purification of water soluble non-electrolytes, such as glycerols, glycols, sugar, formaldehyde; recovery of valuable metals from wastes by chelation; recovery of nicotine from tobacco-dryer gases; in nuclear industries, for processing highly radioactive spent reactor fuels, treatment of radioactive waste and purification of reactor waters. They are used as catalysts in various reactions where acids and alkalis have normally been used as an aid in analytical technique, as plant nutrient; and for the purposes of drying. They are also used in the treatment of industrial waste waters.

0.3 This standard was first published in 1974. The Committee responsible for the preparation of this standard decided to revise it in order to update the same in the light of the experience gained over the last several years. In the revised version, sampling plan has been introduced and modifications have been made in the methods of test for grading, density, void volume, moisture content and operating capacity. In total exchange capacity, the batch method for both the cation exchangers as well as the anion exchangers have been given in detail. Total chloride capacity has been split to include test methods for both strong base and weak base anion-exchange resins, and visual testing given in the earlier version has been excluded.

0.4 Considerable assistance has been drawn from the following ASTM standards published in the Annual Book of ASRM standards, Part 31, 1982:

ASTM D (1782) 31-1978	Operating performance of particulate cation-exchange materials
ASTM D (2187) 31-1977	Physical and chemical properties of particulate ion-exchange resins
ASTM D (2687) 31 1977	Sampling particulate ion-exchange materials
ASTM D (3087) 31-1978	Operating performance of anion-exchange materials for strong acid removal
ASTM D (3375) 31-1975	Column capacity of particulate mixed bed ion-exchange materials

0.5 The Committee took cognizance of the fact that ion-exchange resins are often subjected to tests for attrition (mechanical and chemical), porosity, osmotic shock and bleachable organic content also. The Committee felt that presently in the absence of adequate knowledge of applying laboratory data to plant performance, these test methods were not amenable to standardization. It is hoped that in the near future when more information/data is made available on these methods of tests, they may be stipulated in the Standard.

0.6 Till such time as the product specifications for ion-exchange resins are formulated, the Committee felt that some guidelines should be provided to this effect. Accordingly, a standard format for indenting ion-exchange resins is given in Appendix A which is based on the test methods covered in the standard. In case tests outside this standard are intended to be used, the requirements as well as the method of test shall be subject to mutual agreement between the user and the supplier.

0.7 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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*Rules for rounding off numerical values (*revised*)

1. SCOPE

1.1 This standard prescribes methods of sampling and test for ion-exchange resins.

2. CLASSIFICATION

2.1 Cation Exchange Resins — These are of the following types:

a) Strong acid cation exchanger,

b) Weak acid cation exchanger, and

c) Polyfunctional cation exchanger.

2.2 Anion Exchange Resins — These are of the following types:

a) Strong base anion exchanger,

b) Weak base anion exchanger, and

c) Polyfunctional anion exchanger.

3. SAMPLING OF ION-EXCHANGE MATERIAL FROM PACKAGES OR CONTAINERS

3.1 Sampling from a Single Package and Multiple Package Lots or Shipment — This method is used to sample materials as received from the manufacturer in the original shipping container and prior to any resin-conditioning procedure. Since the ion-exchange material may be supplied in dry or free-flowing state or in moist form it is necessary to employ two different sampling methods.

3.1.1 *Apparatus* —Sampling devices, as shown in Fig. 1 and 2, and sealable, gasproof containers for sample storage, shall be used.

Note — The slotted tube sampler for ion-exchange resins shall consist of two polished brass telescopic tubes with registering slots which are opened or closed by rotation of the inner tube, the outer tube being provided with a point to facilitate penetration.

3.1.2 *Scale of Sampling*

3.1.2.1 *Lot* — All the packages or containers of the same grade of material manufactured at a time shall constitute a lot.

3.1.3 The number of containers or packages shall be selected at random from the lot and shall be in accordance with Table 1. To ensure randomness of selection, procedure given in IS : 4905-1968* may be followed.

TABLE 1 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

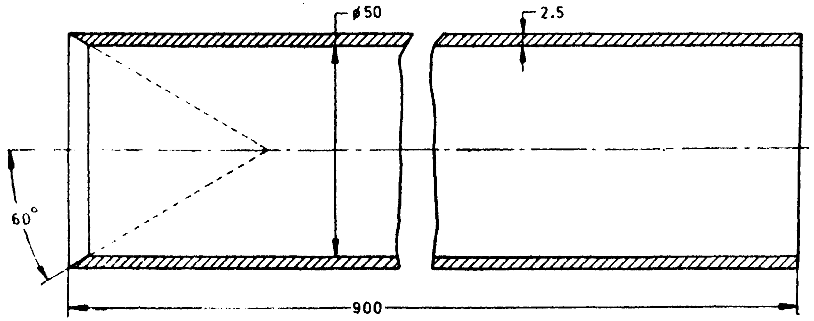
Lot Size (1)	No. of Containers to be Selected (2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 150	7
151 and above	10

Note — In case the lot size is less than 3 containers, each container shall be sampled.

3.1.4 *Sampling Procedure*

3.1.4.1 *Sampling procedure for dry and free-flowing material*

a) If the ion-exchange material is dry or free-flowing and is contained in a drum, rock



All dimensions in millimetres.

Fig. 1 Tube for Sampling of Ion-Exchange Resins

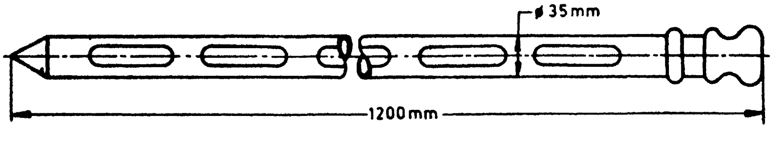


Fig. 2 Slotted Tube for Sampling of Ion-Exchange Resins

the drum slightly from several sides for uniform mixing, and open. The points chosen for probing on the surface should lie on the circle approximately two third the diameter of the drum and should be uniformly spaced.

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*Methods for random sampling.

b) Rotate the inner tube of the sampling device (*see* Fig. 2) to the closed position and thrust the sampling device to the bottom of the drum. Rotate the inner tube to the open position and collect the sample by moving the top of the sampling device in a circle (about 75 to 150 mm in diameter) several times with the open sections forward.

c) Rotate the inner tube to the closed position and remove the sampling device from the drum. Empty the contents into the sample container.

d) Repeat steps (b) through (c) until sufficient sample is obtained. A minimum of three increments is required. Seal the sample container and label.

e) If the ion-exchange material is in a bag, transfer the contents of the bag to a drum which has a diameter less than its height and shall hold the entire contents of the bag and the procedure given in (a) to (d) may be adopted.

3.1.4.2 *Sampling procedure for moist material*

a) If the ion-exchange material is moist and is contained in a drum, invert the drum and allow to stand for 16 hours (overnight) to redistribute any excess water that may be present. Then keep the drum in normal position, rock it slightly from several sides for uniform mixing and open. The points chosen for probing on the surface should lie on a circle approximately two-thirds the diameter of the drum.

b) Rapidly thrust the sampling tube (Fig. 1) to the bottom of the drum. Withdraw the sampling device. Empty the contents into the sample container.

c) Repeat step (b) until sufficient sample is obtained. A minimum of three or four increments is required. Seal the sample container and affix label.

3.2 Sampling from Fixed Bed Ion-Exchange Equipment

3.2.1 This method gives the sampling procedure to obtain a representative sample from an ion-exchange unit under the plant conditions.

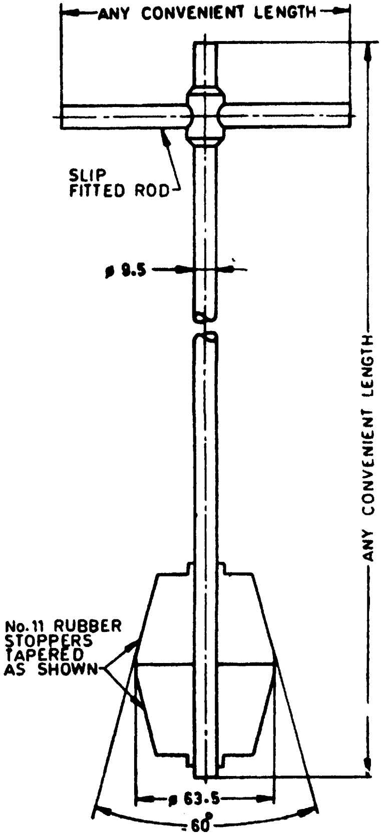
3.2.2 The sampling of the ion-exchange material shall be generally done at the end of the regeneration cycle in order to obtain more information and also to determine the efficiency of regeneration under plant conditions. Determination of metallic, organic or siliceous residues which are not removed in normal plans regeneration, particularly in the case of mixed bed system, is also possible from such sample.

3.2.3 This method will not retrieve ion-exchange material from the bottom 50 mm of a unit without subfill (supporting bed).

3.3 Preparation for Sampling

3.3.1 For mixed bed units containing cation and anion exchange resin, backwash the bed following exhaustion, regenerate, rinse and air mix as in normal operating procedures.

3.3.2 For all other units, backwash the bed following exhaustion, regenerate and rinse as in normal operating procedures.



All dimensions in millimetres.

FIG. 3 SAMPLING ROD

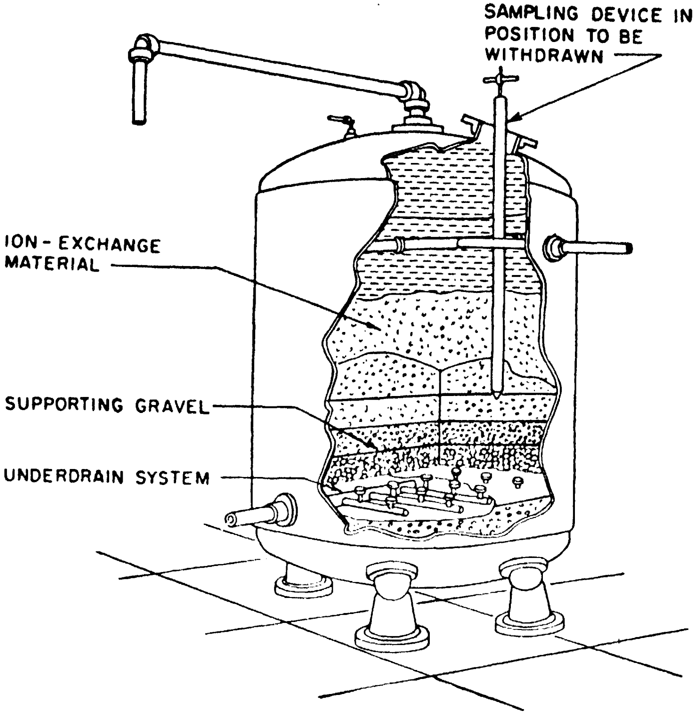


FIG. 4 SAMPLING APPARATUS

3.3.3 Open access opening on top of the ion-exchange equipment. Drain water just to the top of the bed. If the ion-exchange unit has been operating at a temperature above 100°C, it should be cooled to the ambient temperature prior to opening to avoid flashing.

3.4 Sampling Procedure

3.4.1 *Sampling from Fixed Ion-Exchange Equipment having Unrestricted Head Room*

3.4.1.1 *Apparatus* — Sampling devices as shown in Fig. 1, 3 and 4 and sealable gas-proof containers for sample storage shall be used.

3.4.1.2 Select a minimum of 6 points evenly spaced and, if possible, on a circle approximately two-thirds the diameter of the bed. Thrust the sampling rod (Fig. 3) through the ion-exchange material until the subfill or bed support can be felt. Care should be exercised not to damage internal parts, specially in the use of interfacial distributors in mixed bed units.

3.4.1.3 With a gentle up and down motion, slip the sampling tube (Fig. 1) over the top of the sample rod and through the ion-exchange material to seat on the upper conical stopper (Fig. 4). Pull the rod to withdraw the sample tube. Check to make certain that the tube is filled. It may take several attempts to perfect this technique and obtain a full sample tube. Empty the contents into the sample container.

3.4.1.4 Repeat steps 3.4.1.2 through 3.4.1.3 until sufficient sample is obtained. A minimum of 6 increments is required. Seal the sample container and label indicating the following information :

a) Name of the manufacturer,

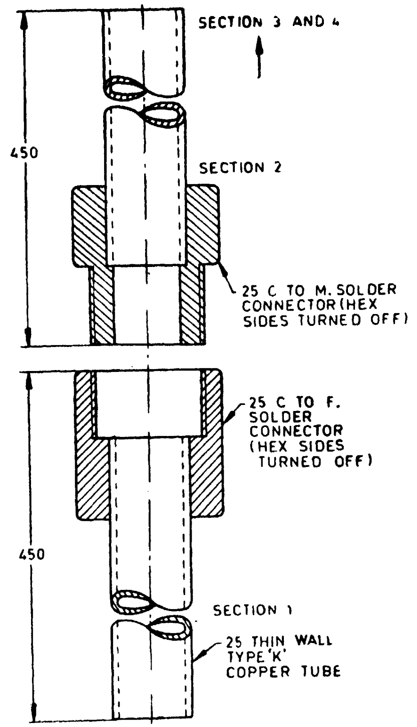
b) Batch number,

c) Date of manufacture,

d) Date and time of sampling, and

e) Type of resin.

3.4.2 *Sampling from Fixed Bed Ion-Exchange Equipment having Restricted Head Room*


SECTION 3 AND 4
450
SECTION 2
25 C TO M. SOLDER CONNECTOR (HEX SIDES TURNED OFF)
25 C TO F. SOLDER CONNECTOR (HEX SIDES TURNED OFF)
450
SECTION 1
25 THIN WALL TYPE 'K' COPPER TUBE

All dimensions in millimetres.

FIG. 5 HOLLOW SAMPLE PROBE GUIDE

3.4.2.1 *Apparatus* — Sampling device as shown in Fig. 5 and 6 and sealable, airtight polyethylene containers for sample storage.

3.4.2.2 Insert the first section of the hollow sample probe guide about half its length into the ion-exchange material. Holding the first section in one hand, screw the second section into the first. Insert the combined sample probe guide about half its combined length into the ion-exchange material. In the same manner, add as many incremental sections as needed to reach within 50 mm of the subfill.

3.4.2.3 Connect one end of the 6 mm soft copper water line to an external source of de-ionized water. This de-ionized water should be of at least equivalent purity to that produced. Insert the free end of the water line into the exposed portion of the hollow sample probe guide and slightly open the external water valve to give a small stream of water. Continue the small flow of water while inserting the water line completely into the hollow sample probe guide. Continue the small flow of water to just suspend the ion-exchange material contained within the hollow sample probe guide.

3.4.2.4 Connect the 9 mm polyethylene tubing to the eduction system suction connection and attach a valved 9 mm water supply to the eduction system power connection. Attach a convenient length of 12 mm outer diameter tubing to discharge connection of the eduction system. Insert the free end of the discharge tubing into a sample container. Large quantities of water will be carried over with the sample. It is convenient to collect the ion-exchange material-water slurry in a very large container.

3.4.2.5 Open the eduction system, supply water and insert the eduction system suction tube into the hollow, sample probe guide to withdraw the sample of ion-exchange material. Continue inserting the suction tube further into the hollow, sample probe guide until all the entrapped ion-exchange material is withdrawn.

3.4.2.6 On complete withdrawal of the whole sample, turn off eduction system water supply and the sample suspension water supply. Remove the suction tube and the 6 mm water line. Remove the hollow sample probe guide by carefully unscrewing each incremental section as it is withdrawn. Close the access opening.

3.4.2.7 If the sample was obtained as a slurry in water, decant the excess water carefully. Transfer all the sample to the final sample container. If a self-dewatering device was used to obtain the sample, carefully transfer all the sample to the final sample container and label.

3.5 Criteria for Conformity — Tests shall be carried out for each important requirement on the individual samples, as stipulated in the relevant specification. For the remaining requirements, tests shall be carried out on the composite sample.

3.5.1 *Individual Samples* — From the test results of each characteristic tested on individual samples, the average $\bar{X}$ and range R shall be calculated as under:

$\bar{X}$ = sum of all the test results on the characteristic divided by the number of test results, and

R = difference between the maximum and the minimum test results.

The lot shall be declared to have met the requirements if the following criteria is satisfied :

a) In case the requirement is specified by a *minimum* limit, the value of the expression $\bar{X} - 0{\cdot}5R \geqslant$ the *minimum* specified limit;

b) In case the requirement is specified by a *maximum* limit, the value of the expression $\bar{X} + 0{\cdot}5\ R \leqslant$ the *maximum* specified limit; and

c) In case the requirement is specified by a range, the value of the expression $\bar{X} + 0{\cdot}5\ R$ lies within the specified range.

3.5.2 *Composite Samples* — In respect of each characteristic tested on the composite sample, the lot shall be declared to have met the requirement if the test result on the composite sample satisfies the corresponding requirement.

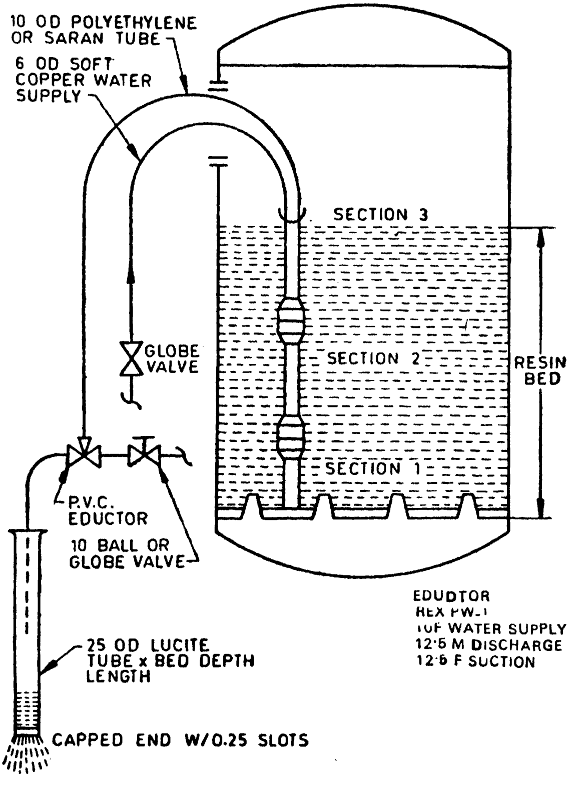
4. GENERAL TESTS

4.1 Resin Pretreatment — Before any examination or characterization of an ion-exchange resin can be performed, the resin shall be pretreated so that it is in the desired ionic form. Unless this pretreatment is carried out, the results obtained may not be representative. The pretreatment may be done with an appropriate regenerant as listed below :

Type of Exchanger	*Regenerant Solution*	*Rinse Requirement*
Strong acid cation	10 percent NaCl/4 percent HCl	Free of Cl^- ion/ neutral to methyl orange
Weak acid cation	4 percent HCL	Neutral to methyl orange
Strong base anion	10 percent NaCl/4 percent NaOH	Free of Cl^- ion/ phenolphthalein alkalinity
Weak base anion	4 percent NaOH	Until 1 drop of H_2SO_4 (0·1 N) neutralizes, 12 ml of effluent

NOTE — In case of mixed bed resin, separate the cation and anion resins by backwashing and pretreat as in **4.1**.

4.1.1 *Procedure* — Place enough resin into a suitable conditioning column to allow the determination of resin characteristics. Backwash with water using a flow rate that will maintain a



All dimensions in millimetres.

FIG. 6 SAMPLING APPARATUS IN PLACE

50 percent expansion of the bed. Continue backwashing until the effluent is clear. Pass about 10 bed volumes of regenerant through the columns at the approximate rate of 0·166 ml/min/ml of sample (contact time of one hour) followed by a rinse with deionized water till the effluent is free of the regenerant.

After rinsing, transfer the sample from the column to a Buchner funnel with a medium porosity filter paper. Fix the funnel over the draining apparatus as shown in Fig. 7. Apply suction to maintain a pressure differential of 40 ± 5 mm Hg below atmospheric pressure. Continue the suction for ten minutes. This drained sample in proper ionic form shall be used for the determination of resin characteristics.

4.2 Grading or Screen Analysis (Wet)

4.2.1 The following set of sieves and pans made of stainless steel conforming to 1·18 mm 850-micron, 600-micron, 500-micron, 425-micron, 355-micron and 300-micron IS Sieves (*see* IS : 460 - 1978*) together with a lid and bottom collecting pan, is used for grading.

4.2.2 Two methods, namely, Method A and Method B have been prescribed. Method B shall be used for routine analysis whereas Method A shall be used as a referee method.

4.2.2.1 *Method A* — A sample of about 100 ml (*see* Note) is placed on top of the screen. The screen is placed on a suitable tray containing demineralized or distilled water, Sieving is done by shaking and vibrating the screen for 15 minutes at 30 to 35 cycles/min keeping the screen all the time inside water. The result is expressed in terms of percentage of total volume of the sample retained on IS Sieve. Repeat the above for each sieve opening. Calculate the percentage retained on each sieve.

NOTE — For measurement of resin volume, transfer the resin into measuring cylinder and tap it to a constant volume.

4.2.2.2 *Method B* — Place the sieve with the largest mesh to be used in a suitable bowl or tray containing about 50 mm depth of de-ionized or distilled water. Place a 100 ml sample into the sieve and agitate the sieve in the horizontal and vertical planes while keeping the mesh under the liquid surface. This should be continued until no undersize particles are found retained on the sieve and should take 10 to 15 minutes. Collect the undersize fraction that has passed and transfer it to the next largest mesh to be used sieving the material in a similar manner as in the first sieve. This process is repeated for the remaining sieves. The result is expressed in terms of percentage of the total original volume of the sample.

Plot a graph on log log paper with sieve opening in millimetres against cumulative percent retained on sieves. Draw the best straight line through the points giving greater weightage to the points representing the largest resin fractions.

4.2.2.3 *Effective size* — Effective size is defined as that sieve opening (in millimetres) which will retain 30 percent of the sample.

4.2.2.4 *Uniformity coefficient* — This is defined as the ratio of sieve openings in millimetres that will retain 40 percent and 90 percent of the total sample.

4.3 Backwashed and Settled Density (BS Density)

4.3.1 *Procedure* — Take about 200 g of the pretreated resin in a graduated column of 25 mm diameter and 1 500 mm length. Backwash with demineralized water for about ten minutes to

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*Specification for IS test sieves.

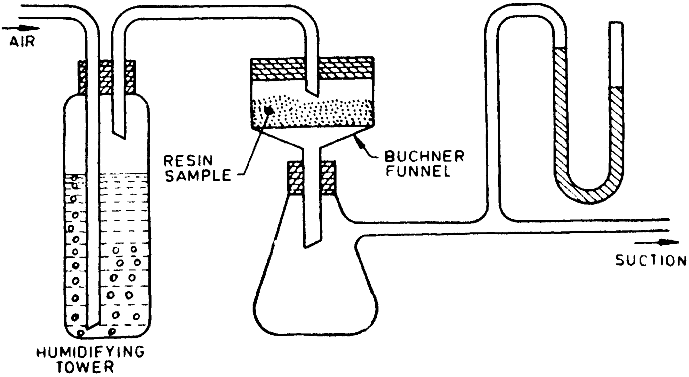

FIG. 7 APPARATUS FOR DRAINING WATER FROM RESIN

maintain a 50 percent expansion of the bed. Allow the bed to settle and then drain at a rate of about 100 ml per minute to within about 25 mm of the top of the bed. Repeat backwashing until two successive readings of volume agree within 5 ml. Record the volume.

4.3.2 *Calculation*

Backwashed and settled

$$\text{density in g/ml, } D_1 = \frac{M}{V}$$

where

M = mass in g of the resin, and

V = volume in ml of the resin.

4.4 Density (Wet Absolute or Hydrated)

4.4.1 *Apparatus*

4.4.1.1 *Glass beaker*

4.4.1.2 *Pyknometer — See* IS : 5717-1970*.

4.4.1.3 *Thermostat — See* IS : 4165-1967†.

4.4.1.4 *Vacuum desiccator — See* IS : 6128-1971‡.

4.4.2 *Procedure* — Weigh accurately 5 g of the pretreated resin correct to 0·000 2 g and transfer it in a dry pyknometer that has already been calibrated with freshly boiled distilled water (*see* IS : 1070-1977§).

The pyknometer is filled to three-fourths of its volume with distilled water and is placed open in the vacuum desiccator for 30 minutes for elimination of air bubbles adhering to the sample. Then, the pyknometer is placed in the thermostat and exposed for one hour to 30°C. Then the level in the pyknometer is raised to the mark by adding distilled water (at a temperature of 30°C) and the pyknometer is weighed with the same accuracy.

4.4.3 *Calculation*

Density (hydrated) in

$$\text{g/ml, } D_2 = \frac{M \times d}{M + M_1 - M_2}$$

where

M = mass in g of the ion-exchange resin,

d = density in g/ml of water,

M_1 = mass in g of the pyknometer filled with water, and

M_2 = mass in g of the pyknometer with water and ion-exchange resin.

4.5 Density (Dry Absolute or Non-Hydrated)

4.5.1 *Procedure* — This determination is the same as described in **4.4.2** except that the ion-exchange resin is dried to constant mass before hand (*see* **4.7.1**).

The density is determined in any non-polar hydrocarbon liquid (preferably xylene), in which the ion-exchange resin does not dissolve or swell. The pyknometer should be calibrated with the same hydrocarbon.

4.5.2 *Calculation*

Density (non-hydrated)

$$\text{in g/ml, } D_3 = \frac{M \times d_1}{M + M_3 - M_4}$$

where

M = mass in g of the ion-exchange resin,

d_1 = density in g/ml of the hydrocarbon,

M_3 = mass in g of the pyknometer filled with hydrocarbon, and

M_4 = mass in g of the pyknometer filled with the hydrocarbon ion-exchange resin.

4.6 Void Volume — The voids in the resin shall be calculated by the following formula :

$$\text{Voids, percent} = \left(1 - \frac{D_1}{D_2} \right) \times 100$$

where

D_1 = backwashed and settled density (BS density) (*see* **4.3.2**), and

D_2 = wet absolute or hydrated density (*see* **4.4.3**).

4.7 Moisture Content

4.7.1 *Procedure* — Weigh about 5 g of the pretreated resin. Dry it in an oven at 105 to 110°C for 16 to 24 hours till a constant mass is obtained. Cool it in desiccator and weigh. If the moisture content of the resin in any other form is required, convert the resin to the desired form with suitable reagents and pretreat as given in **4.1**.

4.7.1.1 *For non-heat resistant resin* — Dry the resin in the vacuum oven at 70°C for about 6 to 8 hours till a constant mass is obtained.

4.7.2 *Calculation*

Moisture content,

$$\text{percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = initial mass in g of the resin, and

M_2 = constant mass in g of the resin after drying.

5. OPERATING CAPACITY

5.1 Cation Exchangers

5.1.1 *Apparatus — See* Fig. 8.

5.1.2 *Reagents*

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*Specification for pyknometers.

†Specification for thermostat for general purpose electric ovens.

‡Specification for desiccators.

§Specification for water for general laboratory use (*second revision*).

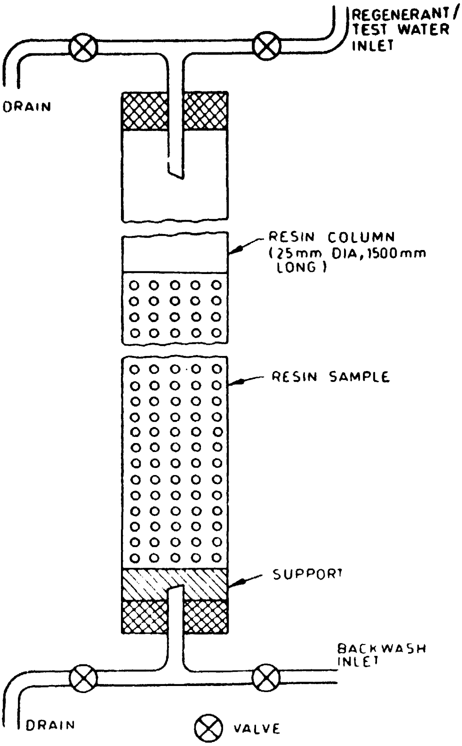

FIG. 8 APPARATUS FOR OPERATING CAPACITY DETERMINATION

5.1.2.1 *Water* — Demineralized water with conductivity less than 5 μS/cm.

5.1.2.2 *Sodium chloride solution* — 100 g/l.

5.1.2.3 *Cation exchange test water* (10 epm).

a) *For sodium cycle method* (*see* **5.1.3**) — Dissolve 0·49 g of $CaCl_2.2H_2O$ and 0·415 g $MgSO_4.7H_2O$ in one litre of water. Adjust the *p*H to 7·5 by Na_2CO_3 (30 g/litre). Hardness of test water shall be 10·0 ± 0·5 epm.

b) *For hydrogen cycle method* (*see* **5.1.4**) — Dissolve 0·21 g $NaHCO_3$ in one litre of water. The alkalinity of the solution will be 2·5 epm. Dissolve in this solution 0·368 g of $CaCl_2.2H_2O$ and 0·311 g of $MgSO_4.7H_2O$ to give a total hardness of 7·5 epm. Total cations shall be 10·0 ± 0·5 epm.

5.1.2.4 *Hydrochloric acid* — 100 g/l.

5.1.2.5 *Sulphuric acid* — 20 g/l.

5.1.3 *Sodium Cycle Method* — This method is used when calcium and magnesium ions are to be removed from water.

5.1.3.1 *Procedure* — Fill the column with sufficient sample in sodium form to give a bed height of 750 mm and maintain a layer of liquid at least 20 mm above the top of the bed at all times. Backwash with water to maintain a 75 percent expansion of the bed till the effluent is clear. Allow the bed to settle and drain at a rate of approximately 100 ml/min until the water level is 20 to 30 mm above the top of the bed. Record the volume in millilitres of the ion-exchange resin. Repeat backwash until two successive readings of volume agree within 5 ml. The average of these two readings will be the sample volume (V_1). Exhaust the resin with test water (*see* **5.1.2.3**) at a flow rate of 0·33 ml/min/ml of exchanger, maintaining a head of liquid more than 50 mm above the top of the bed. Continue the run until the effluent shows 0·2 epm (or other agreed upon hardness level). Record this volume of test water used (V_2).

Pass the regenerant NaCl solution (**5.1.2.2**) through the bed at the rate 0·032 ml/min/ml of sample for 30 min. Rinse the bed with water using the same rate until one bed volume of liquid has been displaced. Increase the rinse rate to approximately 100 ml/min and continue the rinse until the effluent shows 0·2 epm or less hardness.

Repeat the service run as described above. Repeat the cycle beginning with the backwash step till the values obtained in three successive runs agree within ±5 percent of their average capacity.

5.1.3.2 *Calculation*

$$\text{Operating capacity in meq/ml of resin} = \frac{T_H \times V_2}{V_1}$$

where

T_H = hardness of test water, epm [**5.1.2.3** (a)];

V_1 = sample in the bed in ml, and

V_2 = test water used in litres.

5.1.4 *Hydrogen Cycle Method* — This is used when sodium, calcium and magnesium ions are to be removed from water.

5.1.4.1 *Procedure* — Fill the column with H^+ form of resin, backwash, allow to settle and record the volume (V_1) as given in **5.1.3.1**. Exhaust the resin with test water (*see* **5.1.2.3**) at a flow rate of 0·35 ml/min/ml of exchanger, maintaining a head of liquid more than 50 mm above the top of the bed. Continue the run until the free mineral acidity of the effluent has decreased to 0·90 of the theoretical free mineral acidity of the test water. Record the volume of test water used (V_2).

Repeat the backwash and drain as described in **5.1.3.1**.

Pass the regenerant either HCl or H_2SO_4 (*see* **5.1.2.4** and **5.1.2.5**) giving a contact time of 30 min. The flow rates of HCl and H_2SO_4 will be 0·037 and 0·212 ml/min/ml of the exchanger, respectively. Rinse the bed with water using the same rate until one bed volume of liquid has been displaced. Increase the rinse rate to approximately 100 ml/min and rinse for 20 min. Continue the rinse until a 25 ml portion of the effluent shows 10 ppm or less free mineral acidity.

Repeat the service run as described above. Repeat the cycle beginning with the backwash step till the values obtained in three successive runs agree within ± 5 percent of their average capacity.

5.1.4.2 *Calculation*

$$\text{Operating capacity in meq/ml of resin} = \frac{T_c \times V_2}{V_1}$$

where

T_c = total cation, epm [*see* **5.1.2.3** (b)],

V_1 = sample in bed in ml, and

V_2 = test water used in litres.

5.2 Anion Exchangers

5.2.0 *General* — The method can be used to determine the operating capacity of anion exchange materials which are employed for the removal of hydrochloric and sulphuric acid from water.

5.2.1 *Apparatus* — *See* Fig. 8.

5.2.2 *Reagents*

5.2.2.1 *Water* — *See* **5.1.2.1.**

5.2.2.2 *Sodium hydroxide solution (40 g/litre)* — The solution should be freshly prepared to avoid absorption of carbon dioxide from air.

5.2.2.3 *Anion exchange test water (10 epm)* — Add 18·1 ml of sulphuric acid (sp gr 1·84) and 27·5 ml of hydrochloric acid (sp gr 1·19) to 500 ml of water and dilute to 1 litre. Prepare the test water by adding one volume of the mixed acid solution to 99 volumes of water.

5.2.3 *Pretreatment* — Fill the column with resin, backwash and allow to settle as in **5.1.3.1.** Regenerate the sample with NaOH solution for 90 min at a flow rate of 0·11 ml/min for each millilitre of ion-exchanger in the column. Rinse with one bed volume of water at the same flow rate. Increase the rinse rate to 100 ml/min and continue to rinse until 10 bed volumes of water have been used. Backwash, allow to settle and record the volume (V_1) as given in **5.1.3.1.**

Exhaust the resin with the anion exchange test water at a flow rate of 0·33 ml/min/ml of the resin. During the run, test the effluent periodically and continue to run till the resistivity of the effluent is less than 20 000 Ω cm.

5.2.4 *Procedure* — Repeat the backwash and drain as before. Regenerate the sample with NaOH solution maintaining a flow rate of 0·054 ml/min/ml of resin based on regenerated sample volume recorded in the pretreatment step. Continue the flow for 30 min. Rinse the bed with one bed volume of anion exchange test water (**5.2.2.3**) at the same flow rate, then increase the rate to 0·03 ml/min/ml of resin. Determine the effluent resistivity periodically until it is greater than 20 000 Ω cm or any other value agreed upon.

Exhaust the bed by maintaining the flow of test water until the effluent resistivity is less than 20 000 Ω cm. Record the total volume of test water (say, V_2 litres). Repeat the cycle beginning with **5.2.4** till the values obtained in three successive runs agree within ± 5 percent of their average capacity.

5.2.5 *Calculation*

$$\text{Operating capacity in meq/ml of resin} = \frac{T_a \times V_2}{V_1}$$

where

T_a = total anions, epm (**5.2.2.3**), and

V_1 = ml of resin in the bed, and

V_2 = total litres of test water used.

5.3 Mixed Bed Ion-Exchangers

5.3.0 *General* — The method provides for the calculation of capacity in terms of either the volume of water treated to a conductivity end point or to a breakthrough of silica. The test assumes that the cation exchanger has been regenerated to the hydrogen form and the anion exchanger has been regenerated to the hydroxide form. Also, the product tested will contain correct proportions of cation and anion exchange materials.

5.3.1 *Apparatus* — *See* Fig. 8.

5.3.2 *Reagents*

5.3.2.1 *Water* — *see* **5.1.2.1.**

5.3.2.2 *Ion-exchange test water (10 epm)* — Prepare a test water containing in each litre 0·585 g of oven-dried (105 °C) sodium chloride and a sufficient volume of sodium silicate solution to give a silica concentration of 10 ppm as SiO_2.

5.3.3 *Procedure* — Weigh 270 g of the moist, as received, mixed bed ion-exchange material and transfer it into the column in the usual way taking care that no stratification takes place. Maintain the water level in the column about 10 mm over the bed level to minimize air-trapping.

Introduce test water at the top of the column and increase the flow rate to 100 ml/min. Measure and record the effluent conductivity after every 2 litres until a conductivity of 1·0 µs/cm is obtained. Then, record the readings for each 500 ml of the effluent until two successive readings greater than 50 µS/cm (20 000 Ω cm) are obtained. When the effluent conductivity exceeds 1·0 µS/cm, take 50 ml sample for silica analysis. Continue sampling for silica to the conductivity end point, adjusting the effluent volume as necessary to compensate for the sample volume taken. When the conductivity exceeds 50µ S/cm, stop the flow of test water and record the total volume (V) of test water used in litres.

Backwash the bed with water following the same procedure as given in **5.1.3.1** and record the bed volume in millilitres.

5.3.4 *Calculation*

$$\text{Operating capacity to the conductivity end point in meq/ml} = \frac{C \times V}{S}$$

where

C = total electrolytes in test water (epm) which equals the chloride equivalency plus 0·33 silica equivalency,

V = volume of test water used in litres, and

S = volume of the sample tested in ml.

5.3.4.1 *Operating capacity to a silica end point* — Plot the silica content of the effluent in mg/l of SiO_2 as a function of volume throughput. Determine from the curve, the volume (V_s) in litres of effluent at which a silica content of 0·1 mg/litre was reached.

$$\text{Capacity, meq/ml} = \frac{C \times V_s}{S}$$

6. TOTAL EXCHANGE CAPACITY

6.1 Batch Method

6.1.1 *For Cation Exchangers (H^+form)* — Take about 1 g of the pretreated resin in a dry stoppered bottle and add 200 ml of 0·1 N NaOH prepared in 1 N NaCl solution. Mix the contents well in the bottle and keep at room temperature for 16 to 24 hours. Filter the solution through a dry filter paper and take 50 ml of the filtrate for analysis of the alkali content using standard acid, and titrate to methyl orange end point. Calculate the total cation exchange capacity as follows:

6.1.1.1 *Calculation*

$$\text{Total cation exchange capacity in meq/g of dry H}^+\text{form of resin} = \frac{200 \times \text{N NaOH} - 4\,(\text{N} \times \text{ml acid})}{\text{Sample mass (percent solids/100)}}$$

6.1.2 *For Anion Exchanger (Cl^-form)* — Take 1 g of the pretreated resin in a dry stoppered bottle. Add 100 ml of 1 N of $NaNO_3$ solution. Mix well and keep at room temperature for 16 to 24 hours. Filter the solution through a dry filter paper and take 50 ml of the filtrate for the analysis of chloride content using standard $AgNO_3$ solution. Calculate the total anion exchange capacity as follows.

6.1.2.1 *Calculation*

$$\text{Total anion exchange capacity in meq/g of dry Cl-form of resin} = \frac{V \times N \times 2}{M\ \text{(percent solids/100)}}$$

where

V = volume in ml of $AgNO_3$,

N = normality of $AgNO_3$, and

M = mass of sample taken.

6.2 Column Method

6.2.1 *For Strong Acid Group*

a) Place about 25 ml pretreated resin in H^+ form in a 15-mm dia calibrated column.

b) Pass 300 ml of HCl (2N) through the bed at 15 ml per minute.

c) When this acid has drained to resin bed level, rinse the bed with demineralized water at 30 ml/min until the effluent is just alkaline to screened methyl orange.

d) Pass 250 ml of 5 percent NaCl at 8·3 ml/min, collect the effluent and titrate with NaOH (1 N) using screened methyl orange indicator.

e) Further pass 100 ml quantities of 5 percent NaCl at 8·3 ml/min until the titration value on 100 ml is less than 0·2 ml of NaOH (1N) of AR grade.

f) Note the total volume of NaOH (1 N) used.

g) Rinse excess NaCl with demineralized water and measure the tapped volume of resin in sodium form as given in Note under **4.2.2.1**.

6.2.1.1 *Calculation*

$$\text{Strong acid exchange capacity in meq/litre of resin in Na}^+\text{ form} = \frac{\text{Total titre value} \times \text{Normality of NaOH solution} \times 1\,000}{\text{Resin volume in ml}}$$

6.2.2 *For Weak Acid Groups*

6.2.2.1 *Cation-exchange resins having weak acid groups* — The procedure under **6.2.1** (a) to (d) is followed with 1 500 ml of 0·1 N NaOH solution in place of 5 percent NaCl. From the difference of the initial and residual alkali contents, the capacity is calculated as weak acid group capacity in meq/litre of resin in H^+ form.

6.2.2.2 *Cation-exchange resin having both weak acid and strong acid groups (polyfunctional)* — The procedure under **6.2.1** (a) to (d) and **6.2.2.1** are followed separately. Value obtained in **6.2.1** gives capacity due to strong acid groups and difference in values obtained in **6.2.1** and **6.2.2.1** gives the capacity due to weak acid groups.

6.2.2.3 *Cation exchange resin having both carboxylic group and phenolic group* — When the capacities due to individual weak acid groups are to be determined, procedure **6.2.2.1** is followed using NaOH in the first service run and calcium acetate solution (1 N) in the second service run. The capacity realized with calcium acetate solution corresponds to carboxylic acid groups only. The capacity obtained with NaOH solution corresponds to both $COOH^-$ and OH^- (phenolic) groups. The difference between the above two gives the capacity due to phenolic — OH groups.

7. TOTAL CHLORIDE CAPACITY (TCC)

7.1 For Strong Base Anion Exchange Resins

7.1.1 *Apparatus*

7.1.1.1 *Column* — 15 mm diameter.

7.1.2 *Reagents*

7.1.2.1 *Hydrochloric acid solution* — 1 N.

7.1.2.2 *Silver nitrate solution* — 0·1 N.

7.1.2.3 *Sodium nitrate solution* — 1 percent.

7.1.2.4 *Hydrochloric acid solution* — 0·001 N.

7.1.2.5 *Potassium chromate indicator solution*

7.1.3 *Procedure*

a) Place about 30 ml of the resin in 15 mm diameter column and treat with 1 litre of HCl (1 N) at the rate of 20 ml/min. A brief rinse with HCl (0·001 N) is necessary before measuring the volume.

b) Take about 25 ml of the above resin in a column and measure the volume accurately as in **5.1.3.1**.

c) Continue rinsing with HCl (0·001 N) at the rate of 20 ml/min until 100 ml of effluent requires 1·5 ml of silver nitrate (0·1 N) for the chloride titration.

d) Pass 500 ml of 1 percent $NaNO_3$ solution at the rate of 10 ml/min; mix well the effluent collected and titrate 50 ml of it for chloride by one of the standard methods, namely, Mohr's method or mercurimetric method (*see* Appendix **B**). Note this titre value.

e) After a pause of 5 minutes, pass 100 ml of 1 percent $NaNO_3$, collect the effluent and titrate 10 ml against silver nitrate. If more than 0·1 ml of silver nitrate is required, add the titration figure to the result in (d) and pass further 100 ml portions of 1 percent sodium nitrate and check for chloride until the last portion takes less than 0·1 ml of 0·1 N titrating solution.

f) Further pass 100 ml of distilled water at the rate of 20 ml/min.

g) Repeat the procedure (c) to (e) to verify results.

7.1.3.1 *Calculation*

$$\text{Total chloride capacity in meq/litre of resin} = \frac{\text{Total titre in ml} \times \text{Normality of } AgNO_3 \times 1\,000}{\text{Volume of resin in ml}}$$

7.2 For Weak Base Anion Exchange Resins

7.2.0 *General* — Weak base anion resin is generally supplied and used in free base form. The capacity of resin is, therefore, calculated in terms of volume of resin taken in free base form of resin.

7.2.1 *Apparatus*

7.2.1.1 *Column* — 15 mm diameter.

7.2.2 *Reagent*

7.2.2.1 *Sodium hydroxide* — 1 N.

7.2.2.2 *Hydrochloric acid* — 1 N.

7.2.2.3 *Silver nitrate* — 0·1 N.

7.2.2.4 *Sodium nitrate* — 1 percent.

7.2.2.5 *Hydrochloric acid* — 0·001 N.

7.2.2.6 *Potassium chromate indicator*

7.2.3 *Procedure*

a) Place about 30-40 ml of resin in 15 mm diameter column and treat with 1 litre of 1 N NaOH at the rate of 10 ml/min. Rinse with demineralized water free of alkali.

b) Take 25 ml of the above treated resin in column.

c) Convert the resin into chloride form by passing 1 litre of 1 N HCl at the rate of

10 ml/min. Rinse with 0·001 N HCl at the rate of 20 ml/min. Continue rinsing until 100 ml of effluent requires 1·5 ml of 0·1 N $AgNO_3$ for chloride titration.

d) For determination of total chloride capacity, follow the procedure as given in **7.1.3** (d) and (e).

e) Repeat the procedure (c) and (d) to verify the results.

7.2.4 *Calculation*

$$\text{Total chloride capacity in in meq/litre of resin} = \frac{\text{Total titre in ml} \times \text{Normality of } AgNO_3 \times 1\,000}{\text{Volume of resin in ml}}$$

8. STRONG BASE CAPACITY

8.1 *Apparatus*

8.1.1 *Column* — 15 mm diameter.

8.2 Reagents

8.2.1 *Ammonia Solution* — 7 percent (dilute 40 ml liquor ammonia of 0·880 density to 200 ml).

8.2.2 *Sodium Chloride Solution* — 5 percent.

8.2.3 *Silver Nitrate Solution* — 1 percent.

8.2.4 *Potassium Chromate Indicator Solution*

8.3 Procedure

a) Make up 25 ml bed of resin and determine the total chloride capacity as described in **7.1.3**.

b) Completely convert the resin again to the chloride form and rinse.

c) Pass 200 ml of 7 percent NH_3 solution through in 15 minutes.

d) Rinse the column with distilled water at the rate of 20 ml/min until the effluent has the *p*H 8.

e) Then pass 200 ml of 5 percent NaCl through it in 30 minutes and rinse out again the excess chloride with distilled water until 100 ml effluent requires less than 0·5 ml of 0·1 N silver nitrate solution.

f) Pass through 500 ml of 1 percent $NaNO_3$ solution at the rate of 20 ml/min. Collect the effluent, mix well and titrate 50 ml of it for chlorides with $AgNO_3$ (0·1 N) using K_2CrO_4 as the indicator.

g) After a pause of 5 minutes, pass 100 ml of 1 percent $NaNO_3$ solution and titrate the effluent against $AgNO_3$. If more than 0·1 ml of $AgNO_3$ (0·1 N) is required, add the titration figure to the result in (f). Further, pass 100 ml portions of 1 percent $NaNO_3$ solution and check for chloride till the last titre is less than 0·1 ml of 0·1 N titrating solution for 10 ml of effluent.

h) Pass 100 ml distilled water at the rate of 20 ml/min.

j) Repeat the procedure (c) to (h) to verify the results.

8.3.1 *Calculation*

$$\text{Strong base capacity in meq/litre of resin} = \frac{\text{Total titre} \times \text{Normality of } AgNO_3 \times 1\,000}{\text{Volume of resin in ml}}$$

APPENDIX A

(*Clause* 0.6)

STANDARD FORMAT FOR INDENTING ION-EXCHANGE RESINS

A-1. The indentors may specify the actual requirements for their use as per the following parameters:

a) Effective size
b) Uniformity coefficient
c) Fines
d) Backwashed and settled density
e) Wet absolute or hydrated density
f) Dry absolute or non-hydrated density
g) Voids
h) Moisture content
j) Operating capacity
k) Total chloride capacity
m) Total exchange capacity

APPENDIX B

[*Clause* 7.1.3 (d)]

DETERMINATION OF CHLORIDE

B-1. MERCURIMETRIC METHOD

B-1.1 Apparatus

B-1.1.1 *Microburette* — 1 ml or 5 ml with 0·01 ml graduation intervals.

B-1.1.2 *Conical Flask*

B-1.2 Reagents

B-1.2.1 *Diphenylcarbazone Indicator Solution* — Dissolve 0·25 g of C.P. crystalline diphenylcarbazone in a mixture of 95 ml of ethanol and 5 ml of 0·05 N nitric acid.

B-1.2.2 *Bromophenol Blue Indicator Solution* — Dissolve 0·05 g of C.P. crystalline bromophenol blue in a mixture of 95 ml of ethanol and 5 ml of 0·05 N nitric acid.

B-1.2.3 *Dilute Nitric Acid* — 0·05 N.

B-1.2.4 *Sodium Hydroxide Solution* — 1 N.

B-1.2.5 *Standard Mercuric Nitrate Solution* — 0·025 N. Dissolve 4·283 0 g of mercuric nitrate [$Hg(NO_3)_2 \cdot H_2O$] in 50 ml of water, acidified with 0·5 ml of concentrated nitric acid (sp gr 1·42). Dilute the acidified $Hg(NO_3)_2$ solution with water to 1 litre. Filter, if necessary, and standardize against standard sodium chloride solution.

B-1.2.6 *Standard Sodium Chloride Solution* — 0·025 N. Dry some amount of sodium chloride (NaCl) for 1 hour at 600 °C. Dissolve 1·461 3 ± 0·000 2 g of dry salt in distilled water, and dilute to 1 litre at 20 °C in a volumetric flask.

B-1.3 Procedure — Take 50 ml of a neutral solution of the sample containing up to 1·5 mg of chloride in a flask and add 4 drops of bromophenol blue indicator solution. If the solution colour is blue, add dilute nitric acid until a yellow colour is obtained. Then, add sodium hydroxide solution dropwise until a blue colour appears. Again, add nitric acid dropwise to obtain a yellow colour and further add 1·0 ml of nitric acid in excess. Add 10 drops of diphenylcarbazone indicator solution and titrate slowly while stirring with mercuric nitrate solution to the appearance of lilac colour that persists for 30 seconds. Carry out a blank determination with the same amounts of reagents.

B-1.4 Calculation

$$\text{Chloride content, meq/litre} = \frac{(V_1 - V_2) \times N \times 1\,000}{V}$$

where

V_1 = volume in ml of mercuric nitrate solution used for the sample,

V_2 = volume in ml of mercuric nitrate solution used for the blank determination,

N = normality of mercuric nitrate solution, and

V = volume in ml of the liquid taken for test.

B-2. MOHR'S METHOD

B-2.1 Reagents

B-2.1.1 *Standard Sodium Chloride Solution* — 0·1 N.

A-2.1.2 *Silver Nitrate Solution* — 0·1 N. Dissolve 8·496 g of the recrystallized silver nitrate in distilled water and make up to 500 ml in a volumetric flask and shake well. Store it away from direct sunlight in an amber-coloured bottle. Standardization of the normality shall be carried out using 0·1 N standard sodium chloride solution.

B-2.1.3 *Potassium Chromate Indicator Solution* — 5 percent.

B-2.2 Procedure — Transfer 25 ml of the solution into a 250-ml conical flask and add 10 drops of potassium chromate indicator solution. Titrate against standard silver nitrate solution till the reddish brown tinge persists after brisk shaking. Carry out a blank determination.

NOTE 1 — The titration should be carried out in neutral *p*H. If the solution is acidic, acidity may be neutralized using calcium carbonate powder (A R grade).

NOTE 2 — If the solution is alkaline, add slight excess of AR nitric acid and neutralize the excess acidity with calcium carbonate powder (AR grade) before titration.

B-2.3 Calculation

$$\text{Total chlorides (as Cl), meq/litre} = \frac{(V_1 - V_2) \times N \times 1000}{V}$$

where

V_1 = volume in ml of standard silver nitrate solution used in the titration with the material,

V_2 = volume in ml of standard silver nitrate solution used in the blank determination,

N = normality of standard silver nitrate solution, and

V = volume in ml of the liquid taken for the test.

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TO
IS 7330:1988 METHODS OF SAMPLING AND TEST FOR ION-EXCHANGE RESINS

(First Revision)

(*Page* 1, *clause* **0.4**, *line* 3) — Substitute 'ASTM' *for* 'ASRM'.

(*Page* 1, *clause* **0.4**) — Substitute 'ASTM D (2687) 31-1977 Sampling particulate ion-exchange materials' *for* 'ASTM D (2687) 31-1977 Sampling rarticulate ion-exchange materials'.

(*Page* 1, *clause* **0.5**, *line* 4) — Substitute 'leachable' *for* 'bleachable'.

(*Page* 2, *clause* **3.1.1**, *line* 2) — Substitute 'air tight' *for* 'gasproof'.

(*Page* 3, *clause* **3.2.2**, *line* 7) — Substitute 'plant' *for* 'plans'.

(*Page* 4, *clause* **3.4.1.1**, *line* 2) — Substitute 'air tight' *for* 'gas-proof '.

(*Page* 6, *Fig.* 6) — Substitute 'EDUCTOR REX PW-1' *for* 'EDUDTOR REX PW-1'.

(*Page* 7, *clause* **4.2.2.2**, *line* 12) — Substitute 'used for sieving' *for* 'used sieving'.

(*Page* 7, *clause* **4.2.2.3**, *line* 3) — Substitute '90' *for* '30'.

(*Page* 7, *Fig.* 7) — The words 'MERCURY MANOMETER' to be indicated against the manometer shown in the figure.

(*Page* 8, *clause* **4.5.2**, *last line*) — Substitute 'hydrocarbon and ion-exchange resin' *for* 'hydrocarbon ion-exchange resin'.

(*Page* 9, *clause* **5.1.3.1**, *para* 2, *line* 3) — Substitute 'with DM water' *for* 'with water'.

(*Page* 9, *clause* **5.1.4.1**, *line* 5) — Substitute '0.33' *for* '0.35'.

(*Page* 10, *clause* **5.1.4.2**) — Substitute :

$$\frac{`T_c \times V_2\text{'}}{V_1} \quad \textit{for} \quad \begin{matrix} `T_c \times V_2\text{'} \\ V_1 \end{matrix}$$

(*Page* 10, *clause* **5.2.4**, *line* 9) — Substitute '0.33' *for* '0.03'.

(*Page* 11, *clause* 5.3.4, *last line*) — Substitute 'volume of the resin sample' *for* 'volume of the sample'.

[*Page* 12, *clause* 7.1.3(b)] — Substitute the following for the existing:

'b) Measure 25 ml of the above resin as given in 4.2.2.1 (Note) and transfer quantitatively into the column.'

[*Page* 12, *clause* 7.1.3(f)] — Substitute the following for the existing:

'f) Repeat the procedure (a) to (e) to verify the results.'

[*Page* 12, *clause* 7.1.3(g)] — Delete.

(*Page* 12, *clause* 7.1.3.1) — Substitute '10 000' *for* '1 000'.

[*Page* 12, *clause* 7.2.3(b)] — Substitute the following for the existing:

'b) Measure 25 ml of the above resin as given in 4.2.2.1 (Note) and transfer quantitatively into the column.'

[*Page* 13, *clause* 7.2.3(e)] — Substitute '(a)' *for* '(c)'.

(*Page* 13, *clause* 7.2.4) — Substitute '10 000' *for* '1 000'.

(*Page* 13, *clause* 8.2.3) — Substitute '0.1N' *for* '1 percent'.

(*Page* 13, *clause* 8.2.4) — Introduce the following sub-clause after 8.2.4:

'8.2.5 *Sodium Nitrate Solution* — 1 percent'.

[*Page* 13, *clause* 8.3(f), *line* 2] — Substitute '10 ml/min' *for* '20 ml/min'.

[*Page* 13, *clause* 8.3(g), *line* 2] — Substitute 'titrate 10 ml of' *for* 'titrate'.

[*Page* 13, *clause* 8.3(h)] — Substitute the following for the existing:

'h) Repeat the procedure (a) to (g) to verify the results.'

[*Page* 13, *clause* 8.3(j)] — Delete.

(*Page* 13, *clause* 8.3.1) — Substitute '10 000' *for* '1 000'.

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